EXHIBIT C

Sediment Extraction Procedures for PCB Analysis

(USEPA SW-846 Method 3545)

Quantitative Environmental Analysis, LLC

STANDARD OPERATING PROCEDURE (SOP) GEHR3545

1.0 TITLE

General Electric (GE) Hudson River Design Support Sediment Sampling and Analysis Program Standard Operating Procedure for the extraction and cleanup of sediment/solid samples for Polychlorinated Biphenyl (PCB) analysis using the pressurized fluid extraction technique as per SW-846 Method 3545 for subsequent analysis by SW-846 Method 8082.

(Acknowledgement: This SOP is based substantially on internal method SOPs provided by Northeast Analytical, Inc. of Schenectady, N.Y.)

2.0 PURPOSE

The purpose of this SOP is to provide to the chemist the procedures required to perform extractions of PCBs, in sediment/solid samples, using the pressurized fluid extraction technique and to perform the subsequent extract volume reduction and cleanup for the GE Hudson River Design Support Sediment Sampling and Analysis Program.

3.0 SCOPE

The following procedure is utilized by the project laboratories for the extraction and cleanup of PCBs from sediment/solid samples using the pressurized fluid extraction method for subsequent analysis by SW-846 Method 8082.

4.0 COMMENTS

The soxhlet technique may be used in place of the pressurized fluid extraction at the discretion of the supervising chemist.

5.0 SAFETY

The chemist should have received in-house safety training and should know the location of first aid equipment and the emergency spill/clean-up equipment, before handling any apparatus or equipment. Safety glasses and protective exam gloves must be worn when handling glassware and samples. Polychlorinated biphenyls have been tentatively classified as known or suspected carcinogens. The chemist must review the Material Safety Data Sheets (MSDS) for PCBs and all reagents used in the procedure before handling them. All solvents should be handled within a lab fume hood.

6.0 REQUIREMENTS

The chemist must have an understanding of the methods and requirements of USEPA-SW- 846A "Test Methods for Solid Wastes" Volume 1B: Lab Manual, 3rd edition. Methods 3545, 3500B, 3620B, 3660B, 3665A. An approved instructor must also certify the chemist to perform the procedure.

7.0 EQUIPMENT

7.1 Cell Body: ASE 200™ (Accelerated Solvent Extractor) Dionex, 22ML #048821, 33 mL #048822 (or equivalent).

7.2 Cell caps: Dionex #049450 (or equivalent).

7.3 Steel Rod: Used to compresses sample in the cell.

7.4 Hydromatrix (Pre-cleaned and suitable for use): Varian #0019-8004 (or equivalent).

7.5 Metal spatula.

7.6 Mixing Tray: Used to mix sample prior to weighing sample.

7.7 Analytical Balance: Mettler AG-204 (or equivalent) used to determine sample mass.

7.8 Cellulose Filter: Prevents the frits of the cell end pieces from being clogged during ASE extraction.

7.9 Sodium Sulfate: Anhydrous (12-60 Mesh), washed with Hexane and baked overnight at 180°C. Used for the laboratory method blank.

7.10 Hexane: High Purity Solvent Baxter (Burdick/Jackson) #UN1208 (or equivalent).

7.11 Acetone: High Purity Solvent Baxter (Burdick/Jackson) #UN1090 (or equivalent).

7.12 Turbo Vap Evaporator: Zymark #ZW640-3 (or equivalent).

7.13 Turbo Vap Evaporator concentrator tubes: Zymark 250 mL (or equivalent), 0.5 mL endpoint.

7.14 1:1 Hexane/Acetone: 50%/50% by volume solvent mixture prepared in the lab.

7.15 Zymark Turbo Vap LV (or equivalent).

7.16 60 mL VOA vials.

7.17 Vials: glass, 8 dram & 4 dram (with Polyseal sealed cap) (20 mL & 10 mL) capacity, for sample extracts.

7.18 Vial Rack: Plastic rack used to hold vials, during all phases of the extract processing.

7.19 Centrifuge: International Equipment Co., Model CL (or equivalent).

7.20 Wrist Shaker: Burrell wrist action shaker, Model 75 and 88 (or equivalent).

7.21 Florisil: 10% deactivated, solvent washed with 1:1 hexane/ether, baked at 130°C for 16 hours. Deactivated with D.I. water. EM Science #FX0282-1 (or equivalent).

7.22 TBA Reagent: Tetrabutylammonium Hydrogen-Sulfite Reagent (prepared in the laboratory).

7.23 Mercury: Triple distilled Mercury Refining Co, Albany, NY #328502 (or equivalent).

7.24 Sulfuric Acid: H_2SO_4 (concentrated) Mallinkrodt #2468 #UN1830 (or equivalent).

7.25 Pipettes: S/P Disposable Serological Borosilicate Pipettes.

1. 1 mL × 1/10 #P4650-11X (or equivalent)
2. 5 mL × 1/10 #P4650-15 (or equivalent)
3. 10 mL × 1/10 #P4650-110 (or equivalent)

Kimble Pasteur Borosilicate glass pipette 9" #72050 (or equivalent)

7.26 Beakers: Assorted Pyrex: 250 mL, 600 mL, and 1000 mL.

8.0 PROCEDURES

8.1 Sample Preparation

8.1.1 Throughout the entire process it should be noted that if the chemist encounters any problems or difficulties with any samples or steps involved, these problems should be brought to the attention of the supervisor and/or quality assurance manager for guidance and then documented in the extraction logbook.

8.1.2 If the sample is a sediment and contains a water layer, decant and discard the layer as aqueous PCB waste. Mix the sample thoroughly and discard any foreign objects such as sticks, rocks, leaves, twigs, or pebbles. **Note:** however that the sample may be composed entirely of rock, concrete or some other solid material in which case the entire sample is treated as the solid.

8.2 Sample Extraction

8.2.1 Set up one 250-mL glass beaker or 4-oz. jar for each sample. Pick the first sample, label a beaker with the sample number, and tare the beaker. Using a metal spatula, add 10 g to 11 g of the wet sample to the beaker. Samples that are observed to be very wet will require additional mass of sample such that the project sensitivity requirements are met. The moisture content of the sample as determined in Section 8.2.2 should be evaluated so that a larger wet-weight sample can be obtained to provide a dry amount of solids to meet the project sensitivity requirements. The amount taken must consider the size limitations of the ASE extraction cell. The laboratory should target a wet-weight amount of 15 g for very wet samples. Record the weight in the PCB solid extraction logbook to the nearest tenth of a gram. Use the washed and baked sodium sulfate as the sample for the method blank and Laboratory Control Sample (LCS). Record the weight in the sample mass book.

NOTE: ALL SAMPLE CONTAINERS ARE TO BE RETURNED TO THE APPROPRIATE REFRIGERATOR. FOR ALL EMPTY SAMPLE CONTAINERS, SEE THE LABORATORY'S INTERNAL CHEMICAL HYGIENE PLAN FOR PROPER DISPOSAL.

8.2.2 The PCB concentration is to be determined on a dry-weight basis and therefore, the percent total solids must be determined. Weigh approximately 5 grams of the previously homogenized sample in a previously weighed, tarred aluminum-weighing pan. Record the weight of the sample and the tare weight of the pan in the percent total solids log. Place the sample in a drying oven at 100 to 110 degrees Celsius for at least 8 hours. Record the time placed in the oven and the oven temperature in the percent total solids log. Remove the samples from the drying oven and allow to cool in a desiccator. Weigh the pan and sample.

Calculate the percent solids by:

$$\frac{\{(\text{wt. of pan + dried sample}) - (\text{wt.of pan})\}}{(\text{wt. of wet sample})} \times 100\%$$

8.2.3 Before the sample is added to the cell, the sample must be dried. The sample is dried by adding pre-cleaned Hydromatrix. The amount of this drying agent being used depends on how much water is in the sample. The more water present in the sample, the more drying agent will be needed to dry the sample. Mix the sample and drying agent thoroughly with a metal spatula.

8.2.4 The cell size to be used during the extraction will be determined by the final volume of the sample after the drying agent has been added. **Note:** sometimes the sample will have to be separated into two cells for the extraction if to much drying agent has been added.

8.2.5 Select the appropriate cell body size for each sample. Assemble one cell end cap to the cell body. Place 3 cellulose filters into the open end of the cell and push it down to the cell end cap using the black ASE push rod.

8.2.6 Label cells with the sample number. Label the corresponding 60-mL VOA vials on the base of the vial.

8.2.7 Place the cell into a clean mixing pan. Add the dried extract to the cell using the metal spatula to guide the sample into the cell. Any sample that fell outside of the cell will be collected in the mixing tray. Remove the cell from the mixing tray and added the sample that is in the mixing tray to the cell. Compact the sample in the cell, using the steel rod, while the sample is being added. **Note**: rinse the steel rod with acetone and dichloromethane before using on a different sample or placing it in the storage drawer.

8.2.8 Add surrogate and matrix spike solution at this point. The final extract volume concentration of the surrogate compounds tetrachloro-*meta*-xylene (TCMX) and decachlorobiphenyl (DCB) should be 10 ng/mL and

100 ng/mL, respectively. At this time, the GE Hudson River Design Support Sediment Sampling and Analysis Program does not require the preparation and analysis of matrix spike and/or matrix spike duplicate samples. If requested in the future, the final extract volume concentration for the spiked Aroclor (Aroclor-1242) in the matrix spike and matrix spike duplicate sample should be 20,000 ng/mL (**Note:** this spike concentration will require a sample dilution to be performed). The final extract volume concentration of the LCS should be 500 ng/mL.

8.2.9 Assemble the top cell end cap to the cell, hand tighten. Place the first cell to be extracted in position 1 on the cell tray (top tray) and the 60-mL VOA vial in position 1 on the vial tray (bottom tray). The cells and 60-mL VOA vials for each sample must be in the same numerical position on the two trays.

8.2.10 Solvent used for PCB extraction is 1:1 hexane/acetone

8.2.11 Select the appropriate method or schedule for PCB extraction and start the ASE. Recommended ASE extraction conditions for PCB in Sediment are provided in Attachment 4.

8.2.12 When the extraction program is complete, transfer the hexane layer (top layer) using a 10-mL pipette into a pre-rinsed turbo tube or 60-mL VOA vial

if using Turbo Vap LV. Leaving only the water layer in the 60-mL VOA vial. Leave the 10-mL pipette in the turbo tube.

8.2.13 Rinse the 60-mL VOA vial using 5 pipettes of hexane. Hand shake for 30 seconds. Allow the two layers to separate, and pipette the hexane layer, using the same 10-mL pipette, into the turbo tube. Repeat this step 1 more time for a total of 2 hexane extractions on the water layer.

8.2.14 Rinse the 10-mL pipette with two pipettes of hexane on the outside of the 10-mL pipette that was in contact with the sample extract and two pipettes of hexane through the 10-mL pipette and collect into the turbo tube.

8.2.15 All glassware must be rinsed with technical grade (tech)-acetone or a "for rinsing-only" labeled solvent, and dried in the hood before other cleaning steps.

8.3 Solvent Reduction: TurboVap Evaporator System

8.3.1 The Turbo Vap evaporator system is used in place of the Kuderna Danish (KD)-concentrator apparatus. The turbovap uses a heated water bath and positive pressure nitrogen flow/vortex action. The unit maintains a slight equilibrium imbalance between the liquid and gaseous phase of the solvent extract which allows fractional reduction of the solvents without loss of higher boiling point analytes.

8.3.2 Turn the unit on and allow to heat up to the specified temperature for individual solvent use.

8.3.3 As a precaution the TurboVap system regulators should be checked to assure that no residual gas pressure remains within the system and that the gas cylinder valve and gas pressure regulators are both off before placing samples in the apparatus. Residual gas pressure may cause splashing and cross contamination of samples. To bleed the system of residual gas pressure place an empty turbo tube into the water bath and close the lid. Make sure that the nitrogen gas cylinder valve is turned off and slowly turn on the gas pressure regulator. Bleed any residual gas until the regulator output pressure gauge reads "0" psi. Proceed to 8.3.4. Make sure to wipe down all surfaces with hexane before concentration samples.

8.3.4 Place the turbo tube containing the samples into the TurboVap and close the lid. Turn on the gas cylinder valve first and then begin slowly turning the pressure regulator on.

Keep the gas pressure very low, until the solvent level is decreased, to avoid splashing. Increase the gas pressure as the sample reduces maintaining uniform flow throughout the reduction.

8.3.5 Turbo Vap – LV low volume unit: Turn the unit on and allow it to heat up to 38 degrees Celsius. As a precaution the Turbo Vap LV regulator should be checked to assure no residual gas remains in the system. Residual gas may cause splashing and cross contamination of the samples. To resolve this place a vial into the vial in to the turbo vap and close the lif. Press the start button and proceed to turn the gas regulator knob counter-clockwise until the regular reads zero. Place the 60-ml VOA vials into the turbo vap. Press the button to turn on the appropriate row of stations that are being used. The press the start button and adjust the regulator until the samples begin to swirl. Check the sample every few minutes and adjust the gas to keep the samples swirling.

8.3.6 The process for solvent (hexane/acetone) reduction takes approximately 20-30 minutes. Do not leave the unit unattended as extracts may be blown to dryness and PCB loss may occur. Immediately notify a supervisor if an extract is blown to dryness.

8.3.7 Concentrate the solvent to approximately 1.0 mL. Remove the samples from the TurboVap and place in the rack. The remaining solvent will consist largely of hexane since the acetone component is fractionally removed at a faster rate than hexane; however, a solvent exchange with hexane should be completed 3 times to ensure the acetone has been entirely removed. **Note**: Not all samples will evaporate at the same rate; sample extracts containing large amounts of petroleum or other non-volatile liquids may stop reducing

before the 1.0-mL point is achieved. Samples that stop reducing should be removed as soon as possible.

8.3.8 Quantitatively transfer the sample extract with a pasteur pipette into an appropriate volumetric flask (25 mL for soil extracts). Rinse the turbo tube or vial with 3 pasteur pipettes of hexane, then transfer the hexane rinse to the volumetric. Repeat the hexane rinse two more times for a total of three hexane rinses of the turbo tube. After the sample has been transferred, rinse the pasteur pipette with 0.5 mL of hexane into the volumetric flask. Add hexane to the volumetric meniscus mark.

Invert the volumetric flask at least three times to mix completely. Decant the contents into a pre-labeled 8-dram vial.

8.3.9 All dirty glassware must be rinsed with tech-acetone or a "For Rinsing-Only" labeled solvent and dried in the fume hood before being washed.

8.4 Sample Extract Cleanup

8.4.1 Most extracts of environmental samples that are to be analyzed for PCBs by gas chromatography with electron capture detection contain co-extracted xenobiotics and other interfering substances which must be removed before accurate chromatographic analysis can be performed.

8.4.2 Sulfuric acid, sulfur removal and Florisil® clean-ups should be performed on every sample. The sequence and number of replicates of cleanup steps performed are recorded by the sample preparation chemist on the sample tracking log sheet. Sample extract cleanups are performed on set volume extracts. The set volume is 25 mL for sediment/solid samples.

8.4.3 Sulfuric Acid Wash

8.4.3.1 The concentrated sulfuric acid treatment removes hydrocarbons and other organic compounds, which are co-extracted with the PCB residues.

8.4.3.2 Chill the sample to approximately 0°C. Add 5.0 mL of concentrated H_2SO_4 and shake for 30 seconds by hand, centrifuge for approximately 1 minute, transfer approximately 20 mL of the hexane upper layer to an 8-dram vial.

8.4.3.3 Repeat 8.4.3.2 if the sample extract appears to be heavily loaded (opaque) with colored material. Two to three acid washes may be required. **Note**: it is entirely possible that all colored material will not be removed from the extract.

8.4.4 Elemental Sulfur Clean-up

8.4.4.1 Elemental sulfur is soluble in the extract solvents used for sediment and soil samples. It is commonly found in many sediment/soil samples, decaying organic material, and some industrial wastes.

Large amounts of sulfur can cause the electron capture detector (ECD) to signal saturate for long periods during the elution envelope of PCBs. Even small amounts of sulfur can interfere with PCB measurement as a co-eluting chromatographic peak.

8.4.4.2 Two techniques exist for the elimination of elemental sulfur in PCB extracts. Mercuric precipitation (Mercury Shake) and the Tetrabutylammonium (TBA) sulfite procedure.

Tetrabutylammonium sulfite causes the least amount of degradation to a broad range of pesticides and organics compounds, while mercury may degrade organophosphorus and some organochlorine pesticides. The TBA procedure also has a higher capacity for samples containing high concentrations of elemental sulfur.

8.4.5 Removal of Sulfur Using Mercury

8.4.5.1 Mercury is a highly toxic metal. All operations involving mercury should be performed within a hood. Prior to using mercury, the chemist should become acquainted with proper handling and emergency spill/clean-up procedures associated with this metal and must have reviewed the material safety data sheet for mercury.

8.4.5.2 Add 1-3 drops of mercury to the sample extracts, cap, and place on the wrist shaker for 30 minutes. The sulfur is converted to mercuric sulfide and precipitates out of the sample extract. A black precipitate may be seen in sample extracts containing elemental sulfur.

8.4.5.3 Remove the sample extracts from the wrist shaker and place in the centrifuge at a setting and duration appropriate to spin down the solids.

8.4.5.4 Transfer the sample extract to a clean 8-dram vial.

8.4.5.5 The precipitated sulfur can be removed from the extract by performing a sulfuric acid clean-up or a Florisil® slurry.

8.4.6 Removal of Sulfur using TBA Sulfite

8.4.6.1 The TBA procedure removes elemental sulfur by conversion to the thiosulfate ion, which is water-soluble.

8.4.6.2 Add 2.0 mL TBA Sulfite Reagent, 1.0 mL 2-propanol, and approximately 0.65 g of sodium sulfite crystals to the extract and shake for at least 5 minutes on the wrist shaker and observe. An excess of sodium sulfite must remain in the sample extract during the procedure. If the sodium sulfite crystals are entirely consumed add one or two more aliquots (approximately 0.65 g) to the extract and observe.

8.4.6.3 Place the samples on the wrist shaker for 45 minutes observing at 15-minute intervals to make sure that the sodium sulfite is not consumed. Add 5 mL organic free water and shake for 10-15 minutes.

8.4.6.4 Place the samples into the centrifuge and spin at a setting and duration appropriate to spin down the solids.

8.4.6.5 Transfer the hexane layer to a new 8-dram vial and cap.

8.4.7 Florisil® Adsorption (Slurry)

8.4.7.1 The Florisil® slurry removes co-extracted polar compounds, residual water, and residual acid and is recommended as the final cleanup step before the extract is submitted for GC analysis.

8.4.7.2 Add approximately 3 grams of tested and approved 10% deactivated Florisil® to each vial containing the sample extract.

8.4.7.3 Vigorously shake the vial for approximately 1 minute by hand or on the wrist shaker.

8.4.7.4 Place the vial(s) into the centrifuge at a setting and duration appropriate to spin down the solids.

8.4.7.5 Transfer the extract to a clean 8-dram vial.

8.5 Extract Screening and Dilution

8.5.1 Screening of PCB extracts by GC to determine the approximate concentration before final analysis is highly recommended. If possible, prior site history and estimates of sample concentration will be provided by field personnel and may be used to determine what, if any, extract dilution is necessary.

8.5.2 The supervising chemist is responsible for determining initial screening dilutions. Extract dilutions are prepared by transferring an aliquot of the original sample extract into a vial containing the correct amount of "make up" volume of hexane. Dilutions must be recorded in the instrument logbook or in the data system.

8.5.3 Perform the dilution using an appropriate disposable volumetric pipette to transfer the extract and for the make-up volume of hexane. Make sure that the vial is properly labeled. Cap and invert the vial at least three times to thoroughly mix the extract with the solvent.

8.5.4 Transfer 1 mL of the extract to a labeled 1.5-mL GC autosampler vial. Record the sample data and submit with the sample extracts to the GC analyst.

9.0 QUALITY CONTROL

9.1 This section outlines the necessary quality control samples that need to be instituted at the time of sample extraction. The data from these quality control samples is maintained to document the quality of the data generated.

9.2 With each batch of samples to be extracted a method blank is processed. The method blank is carried through all stages of sample preparation steps (including clean-up steps). For sediment/solid samples, a laboratory sodium sulfate blank is processed.

9.3 At this time, the GE Hudson River Design Support Sediment Sampling and Analysis Program does not require the preparation and analysis of matrix spike and/or matrix spike duplicate samples. If requested in the future, a matrix spike for Aroclor-1242 is to be analyzed at a rate of 1 matrix spike per every 20 samples at a concentration of 20,000 ng/mL in the extract (**Note:** this spike concentration will require a sample dilution to be performed). Also a matrix spike duplicate sample is to be analyzed at a rate of 1 per every 20 samples.

9.4 A QC reference check standard (LCS) is also prepared and analyzed for Aroclor-1242 at a concentration of 500 ng/mL in the extract. For sediment/solid samples, sodium sulfate is used.

9.5 Surrogate compounds are added to each sample, matrix spike, matrix spike duplicates, method blank, and QC reference check standard (LCS) at time of extraction. The surrogate compounds TCMX and DCB are to be added for final extract concentrations of 10 ng/mL and 100 ng/mL, respectively.

10.0 REFERENCES

1. U.S. EPA SW-846 "Test Methods for Evaluating Solid Waste; Volume 1B Laboratory Manual Physical/Chemical Methods", Office of Solid Waste and Emergency Response, Third Edition, Final Update III, December 1996.

2. "Guide to Environmental Analytical Methods", Third Edition, Genium Publishing Corporation, 1996.

11.0 ATTACHMENTS

1. ASE Methods

2. ASE control panel keypad.

3. ASE cell cleanup procedure.

4. Recommended ASE Extraction Conditions for PCB in Sediment

ATTACHMENT 1

ASE METHODS

METHOD #	ANALYTE OF INTEREST	MATRIX	REQUIRED SOLVENT *	AMOUNT OF SAMPLE
1	PCB	WIPE	1	WIPE
2	PET I.D.	SOIL	1	10 g
2	8270	SOIL	3	30 g
3	**PCB**	**SOIL**	**2**	**10 g**
4	NONE	NONE	2	NONE
5	**PCB (RUSH)**	**SOIL**	**2**	**10 g**

*REQUIRED SOLVENT CHART

1 - HEXANE
2 - 1:1 HEXANE / ACETONE
3 - 1:1 DICHLOROMETHANE / ACETONE

ATTACHMENT 2
CONTROL PANEL KEYPAD

* TRAY	Tray is in free spin for manual turning.
TRAY *	Tray drive mechanisms are engaged and cannot be moved manually.
RINSE	Starts a manual rinse cycle.
* START	System is idle.
START *	system is currently running a method or schedule.
ABORT	Interrupts current run. Continue with abort function to terminate ASE run.
MENU	Displays a list of available screens.

* - LIGHT IS ON

ATTACHMENT 3
ASE cell cleanup procedure

Remove the end caps of ASE cells. Using a metal spatula designated for cell clean up, push the extracted sample out of the cell into a garbage can. Wash the interior and exterior of the cell and cell end caps with soap and water (use the brush designated for ASE use only). Dry the cell parts with a paper towel and reassemble the cell.

Run the washed cells on the ASE (use a new 60-mL VOA vial for each cell) using method 7 for 22 mL cells, 8 for 33 mL cells and 1:1 dichloromethane/acetone as the solvent.

Note: After the cells has been used 20 times or if the frits become clogged, the cell end caps should be taken apart and sonicated for 10 minutes in acetone and 10 minutes in dichloromethane.

ATTACHMENT 4
Recommended ASE Extraction Conditions for PCB in Sediment

The following instrument conditions will be utilized for extraction of sediment samples by accelerated solvent extraction for PCB. These conditions may need to be optimized, as needed, according to the instrument manufacturer's recommendations. Once conditions are established, the same procedures should be performed on all samples.

Recommended extraction conditions for Dionex ASE200

Oven Temperature: 150°C
Pressure: 1750 psi
Static time: 7 minutes (Instrument will automatically perform a 7-minute pre-heat equilibration cycle)
Flush volume: 60% of cell volume
Nitrogen Purge: 180 seconds at 150 psi
Static Cycles: 3